Updated Trends in Materials' Outgassing Technology

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History

- Predessor Joe Colony reported on chemical analysis from GSFC chambers in 1979 in NASA TM 80585
- TM covered 8 years of reports from 1970-1978
- Utilized FORTRAN source code on 80 deck cards to extract information out of each report to create the data bank.
- Looked to identify common outgassing chemical species from flight hardware, spacecraft, and ground support equipment (GSE) from Thermal vacuum exposure.
- Tallied 102 different chemical species from 1163 reports.
- Typically only reviewed data from Cold Fingers and when depositions tallied above 10 mg.
- Together with Fred Gross developed library of FTIR plots and mass spectrum data from GC.

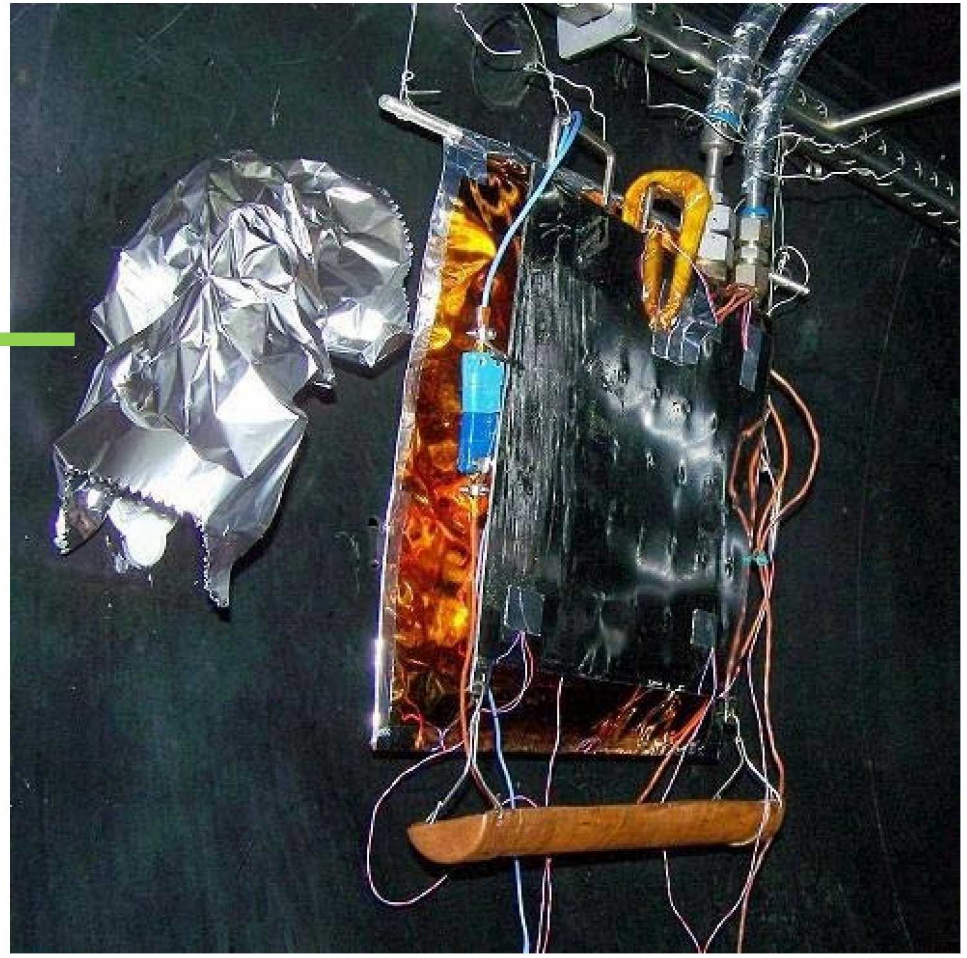
Updated Trends

- Chemical Analysis reports (CARs) are created in MS Word by GSFC Materials Engineering Branch, Code 541, and are electronically mailed to various project personnel.
  - Cut and pasted chemical species as text into rows into Excel spreadsheet and hand entered other pertinent information report.
- Only reviewed CARs that I had received and that had the Scavenger Plate (SP) or the chamber's cryocoil activated.
  - covered ~3 years of reports from late 2007 - mid 2010, tallying 152 reports out of 713 total reports with >100 identified species.
  - No residue limitations.
- Utilized database of reports to identify common outgassing chemical species from flight hardware, spacecraft, and ground support equipment (GSE).

Chamber Mass Collectors

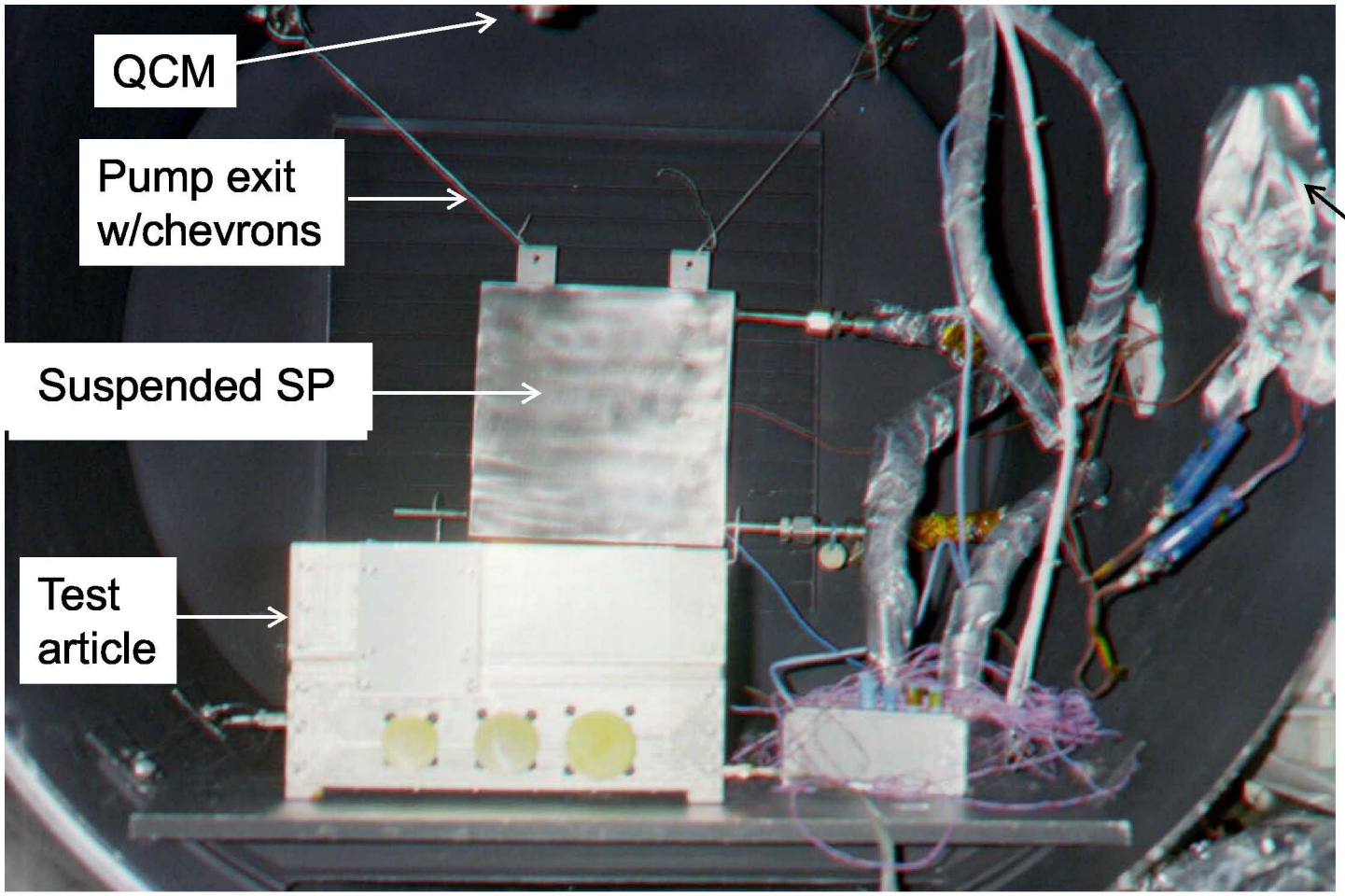
- All GSFC TV chambers have a Cold Finger (CF)
  - Typically ~16 in^2 of active area, with drain
  - Liquid Nitrogen (LN2) flows through at end of test, typically after hardware had achieved desired test criteria, for an 8 hour duration
- Most GSFC TV chambers have a Scavenger Plate (SP)
  - Most are 1 foot square with both sides active
  - LN2 flows through the SP continuously during TV testing of article.
  - Has a trough mounted underneath to collect excess residue.
- 2 GSFC Chambers have cryocoils in the door.
  - Assists with "pumping" the chamber down to high vacuum.
  - Are not insulated and are several feet in total length.
  - LN2 flows through coils continuously, acts like a SP.
  - Chamber 245 and 281
- Chamber vacuum pump
  - Most chambers have cryopumps that have chevrons in front of pumping exit.
- Quartz Crystal Microbalances (QCMs)
  - Mounted on flexible fluid lines; able to be moved anywhere in TV chamber
  - Able to be set at any temperature, typically -20C.

Chamber Mass Collectors



Typical CF and SP location inside chamber

Chamber Mass Collectors



Sample collection

- Residue samples are rinsed off SP and CF, collected in clean/furnace baked collection bottles.
- Rinsate is typically Spectra or Optima-grade IPA or chloroform.
- Sample is evaporated on foil dish and weighed on 5 decimal place scale (0.01 mg)

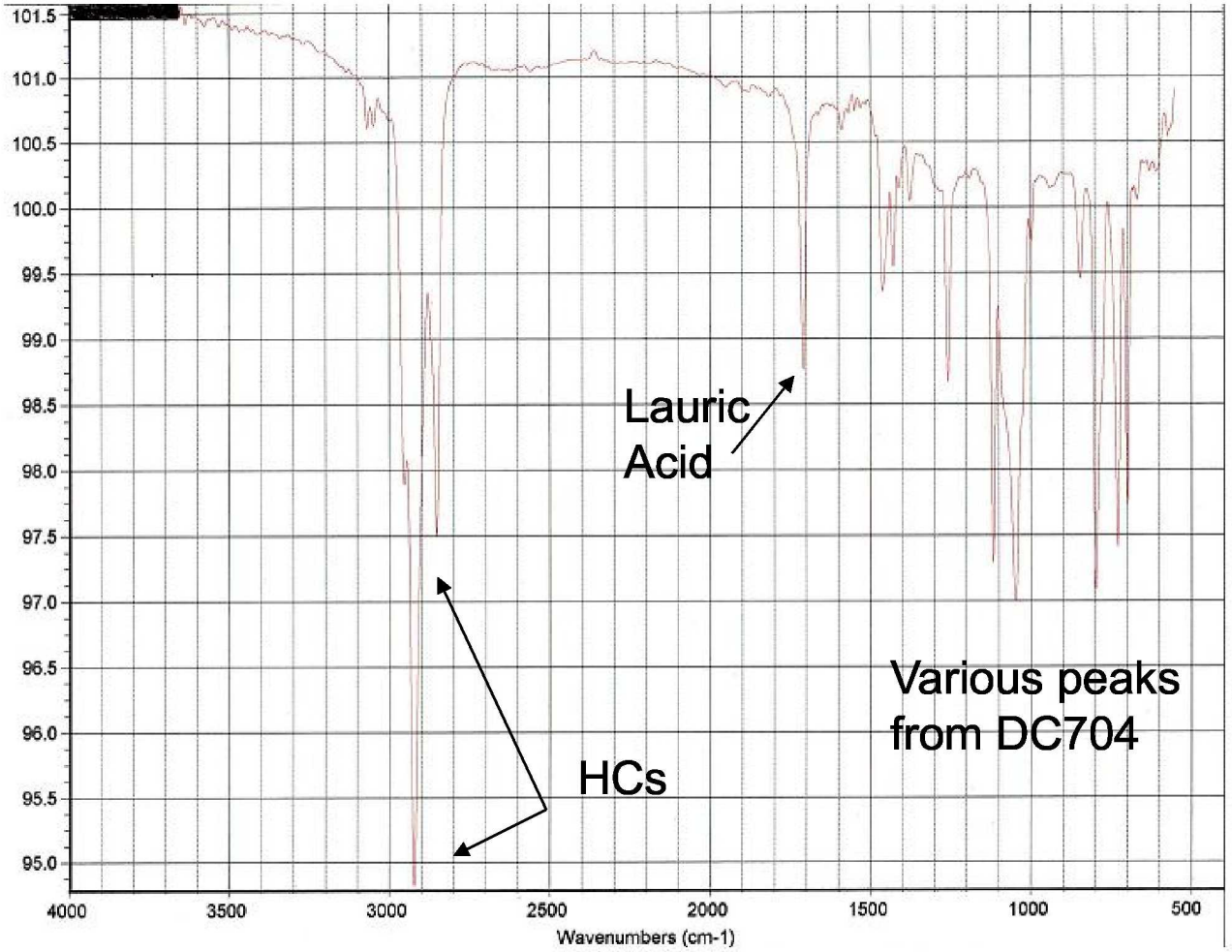
Chemical Analyzers

Fourier Transform Infra-Red (FTIR) Spectrometer

- Sample placed on ZnSe crystal
- Scans from 2.5 to 20 microns (4000 to 500 cm^{-1})
- Takes about 10 minutes per scan, plots in a minute or 2.
- Can File Save scan and compare with library spectra to identify species
- More qualitative analysis, but can yield quantitative estimates
- An older Nicolet 380 apparatus, from Thermo Scientific, is shown on right.

Chemical Analyzers

- FTIR scan (Hydrocarbons, Lauric acid, DC704 peaks)



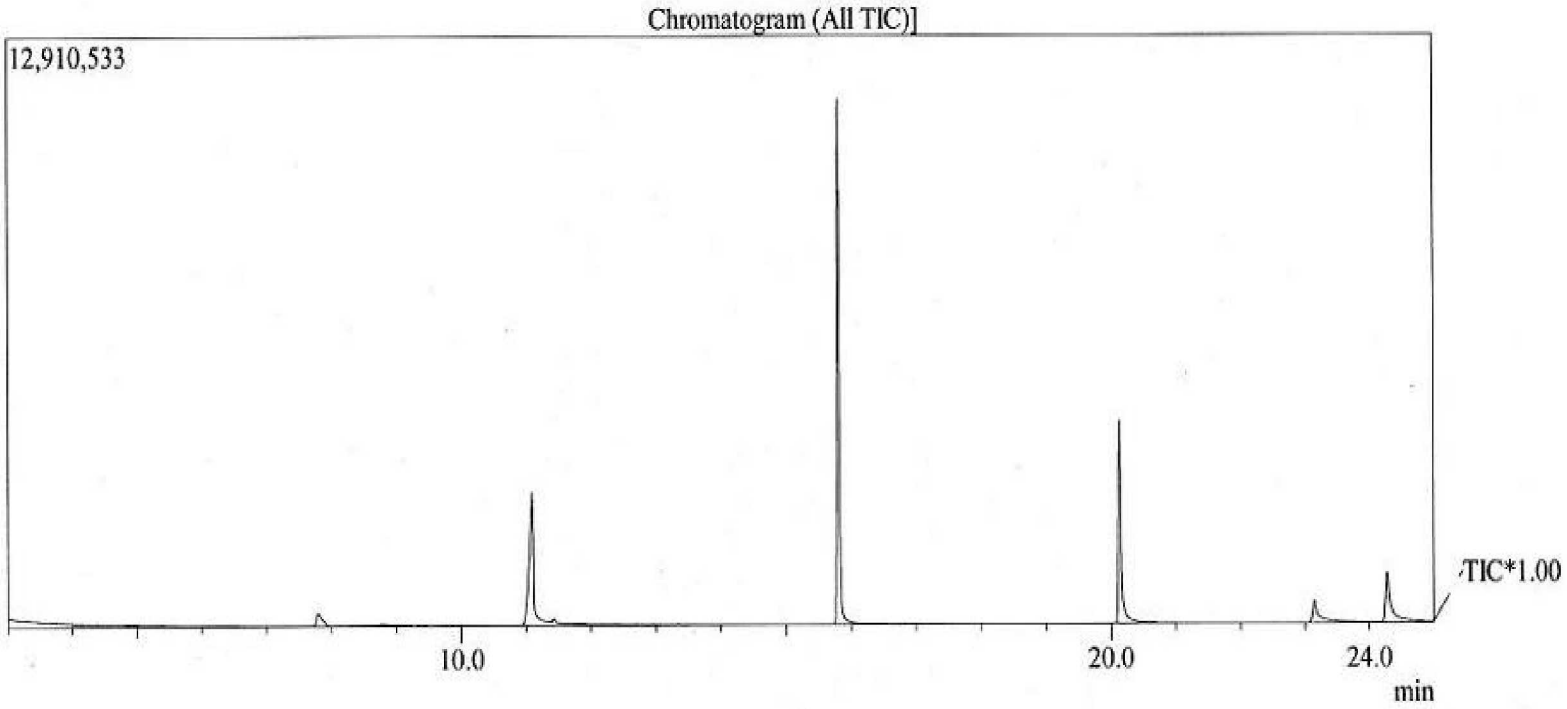
Chemical Analyzers

Gas Chromatograph/Mass Spectrometer (GC/MS)

- Requires UHP Helium, Slight vacuum pulled internally
- Sample introduced as liquid (1 microliter)
- Thermal extraction
- Ramped rate from 25C to 300C
- Takes 25 minutes/scan
- More post processing Involved to ID species
- Shimadzu GC-2010 shown

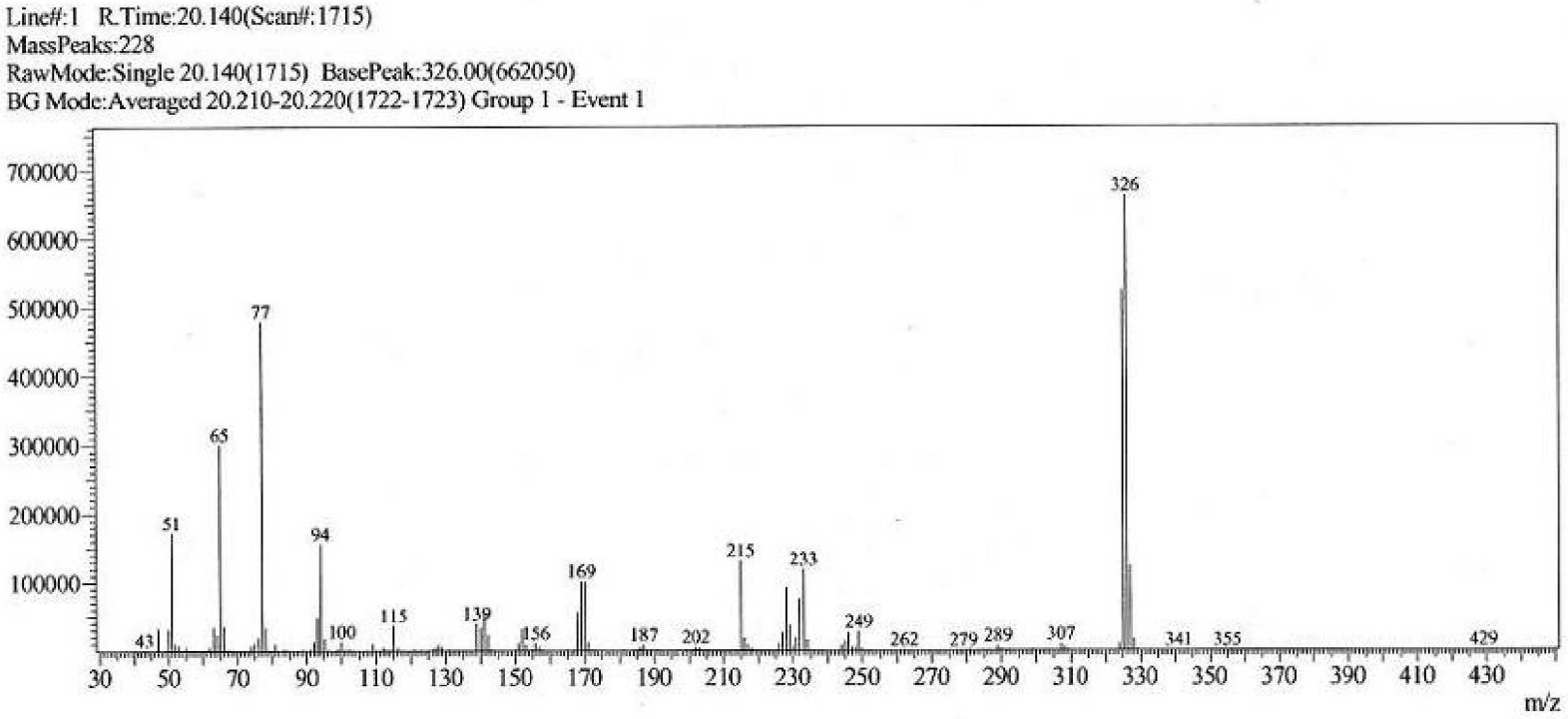
Chemical Analyzers

- GC output scan, species fragment/outgas at specific temps, which is related to a retention time.
- Lighter more volatile species peak early, heavier weighted molecules peak later (higher temp)



Chemical Analyzers

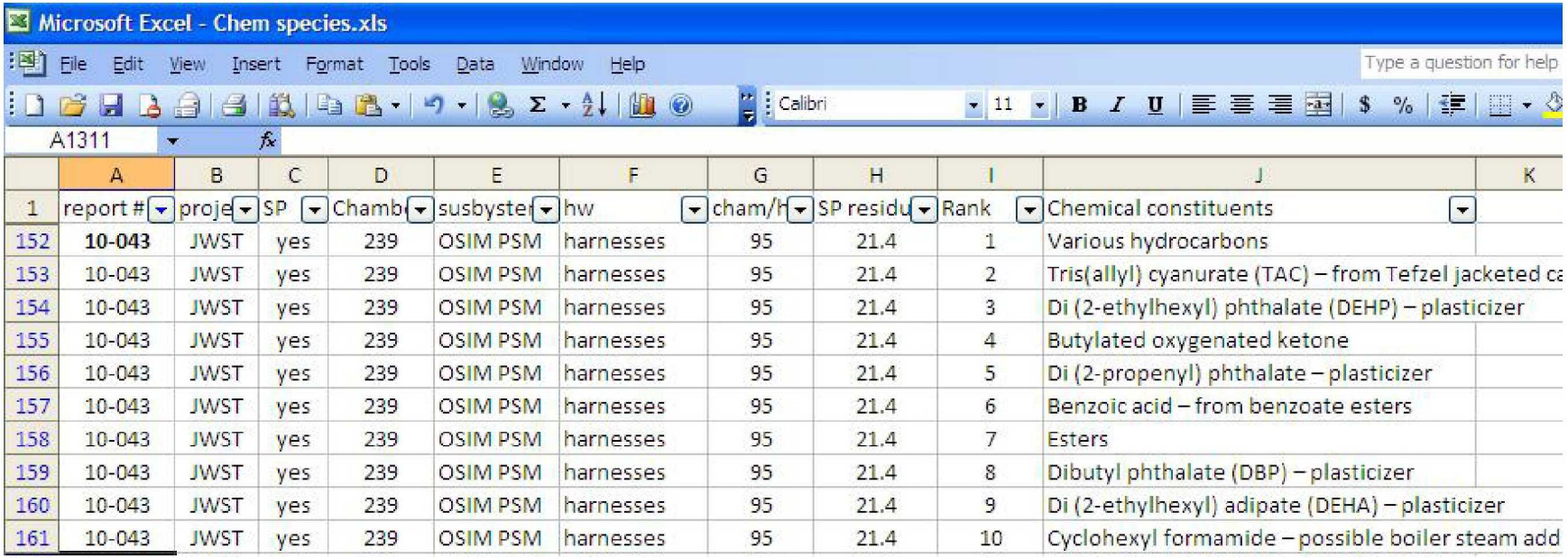
- Mass Spectrum output scan from a selected peak at time 20.14 minutes.
- Primary peak at 326 m/z with secondary peak at 77 m/z yielded triphenyl phosphate (TPP).



Database inputs

- Data extracted from a GSFC Chemical Analysis report (CAR) and insert into an Excel spreadsheet
- Column 1-Test report number
- Column 2-Spacecraft Project acronym
- Column 3-Scavenger plate or Cryocoil
- Column 4-Chamber (located in GSFC Bldg 7)
- Column 5-Subsystem of project hardware
- Column 6-generalized test article
- Column 7-Chamber/Hardware temperature (deg C)
- Column 8-Residue collected in milligrams (mg)
- Column 9-Chemical Specie rank in report (1-10)
- Column 10-Chemical specie

Database CAR listing



Microsoft Excel - Chem species.xls

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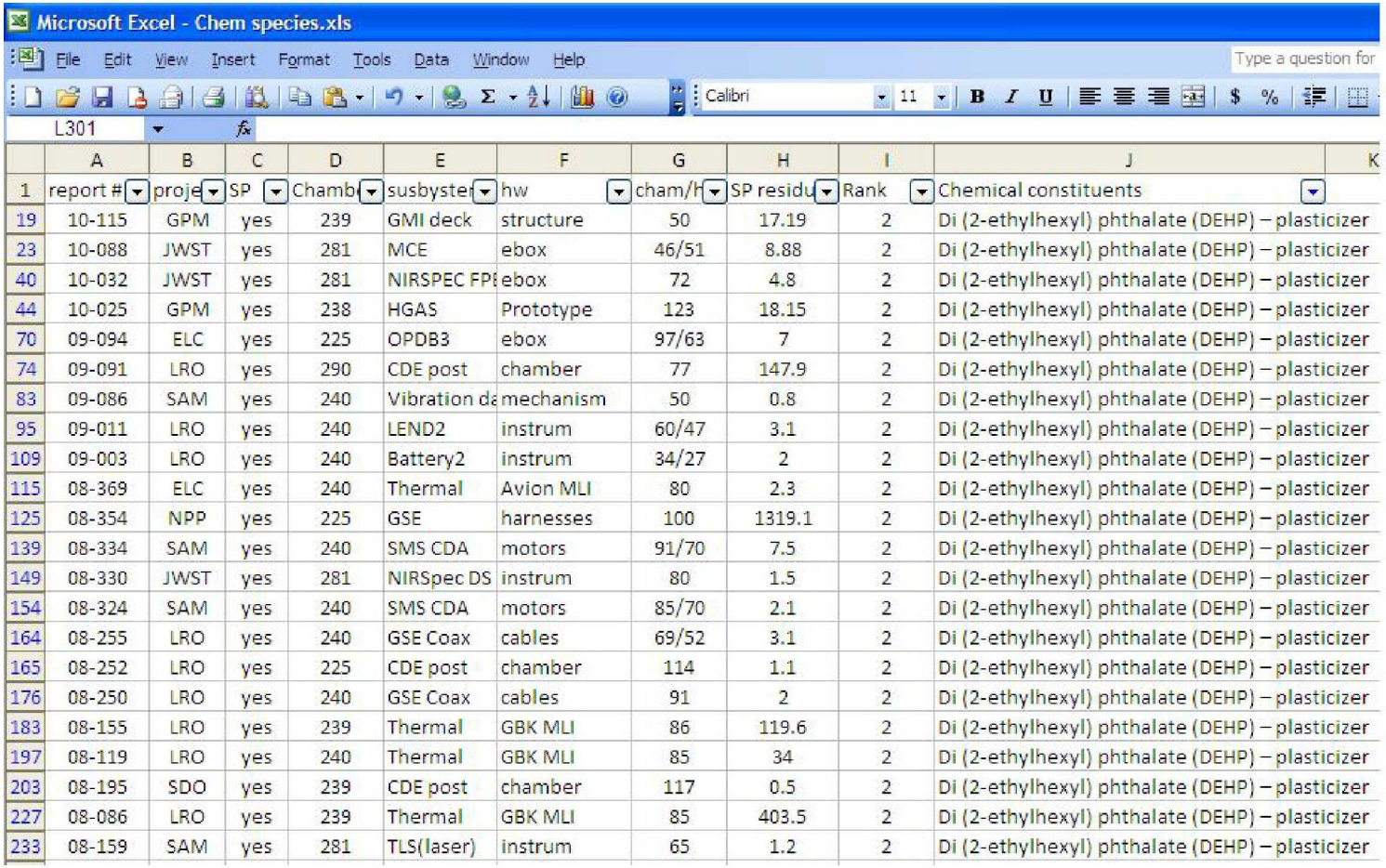
A1311

	A	B	C	D	E	F	G	H	I	J	K
1	report #	proje	SP	Chamb	susbyste	hw	cham/h	SP residu	Rank	Chemical constituents	
152	**10-043**	JWST	yes	239	OSIM PSM	harnesses	95	21.4	1	Various hydrocarbons	
153	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	2	Tris(allyl) cyanurate (TAC) – from Tefzel jacketed ca	
154	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	3	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
155	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	4	Butylated oxygenated ketone	
156	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	5	Di (2-propenyl) phthalate – plasticizer	
157	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	6	Benzoic acid – from benzoate esters	
158	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	7	Esters	
159	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	8	Dibutyl phthalate (DBP) – plasticizer	
160	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	9	Di (2-ethylhexyl) adipate (DEHA) – plasticizer	
161	10-043	JWST	yes	239	OSIM PSM	harnesses	95	21.4	10	Cyclohexyl formamide – possible boiler steam add	

Copied species as text into Excel, hand entered single line of other CAR info and then created a Sort header

Database DEHP listing



Microsoft Excel - Chem species.xls

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	A	B	C	D	E	F	G	H	I	J	K
1	report #	proje	SP	Chamb	susbyste	hw	cham/h	SP residu	Rank	Chemical constituents	
19	10-115	GPM	yes	239	GMI deck	structure	50	17.19	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
23	10-088	JWST	yes	281	MCE	ebox	46/51	8.88	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
40	10-032	JWST	yes	281	NIRSPEC FPE	ebox	72	4.8	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
44	10-025	GPM	yes	238	HGAS	Prototype	123	18.15	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
70	09-094	ELC	yes	225	OPDB3	ebox	97/63	7	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
74	09-091	LRO	yes	290	CDE post	chamber	77	147.9	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
83	09-086	SAM	yes	240	Vibration da	mechanism	50	0.8	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
95	09-011	LRO	yes	240	LEND2	instrum	60/47	3.1	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
109	09-003	LRO	yes	240	Battery2	instrum	34/27	2	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
115	08-369	ELC	yes	240	Thermal	Avion MLI	80	2.3	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
125	08-354	NPP	yes	225	GSE	harnesses	100	1319.1	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
139	08-334	SAM	yes	240	SMS CDA	motors	91/70	7.5	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
149	08-330	JWST	yes	281	NIRSpec DS	instrum	80	1.5	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
154	08-324	SAM	yes	240	SMS CDA	motors	85/70	2.1	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
164	08-255	LRO	yes	240	GSE Coax	cables	69/52	3.1	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
165	08-252	LRO	yes	225	CDE post	chamber	114	1.1	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
176	08-250	LRO	yes	240	GSE Coax	cables	91	2	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
183	08-155	LRO	yes	239	Thermal	GBK MLI	86	119.6	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
197	08-119	LRO	yes	240	Thermal	GBK MLI	85	34	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
203	08-195	SDO	yes	239	CDE post	chamber	117	0.5	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
227	08-086	LRO	yes	239	Thermal	GBK MLI	85	403.5	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	
233	08-159	SAM	yes	281	TLS(laser)	instrum	65	1.2	2	Di (2-ethylhexyl) phthalate (DEHP) – plasticizer	

Sorted by chemical species and rank, newest to oldest report #

1979 Top 20

1. ALHYD-Aliphatic Hydrocarbons (185)
2. MESIL-Methyl Silicones (129)
3. DEHP-Di (2-ethyl hexyl) phthalate (114)
4. ESTER-Esters (100)
5. DBP-Dibutyl phthalate (27)
6. PHEST-Phosphate esters (24)
7. ARMHY-Aromatic Hydrocarbons (23)
8. MPHSI-Methyl Phenyl Silicones (19)
9. URETH-Urethanes (18)
10. HMBZP-Hydroxy Methoxy Benzophenones-antioxidant (17)
11. BHT-Butylated Hydroxy Toulene(16)
12. DC704-DC 704 Diffusion Pump Oil (15)
13. RTV56-Methyl Phenyl Silicones from RTV-560 or -566 (12)
14. ORGAC- organic or fatty acids (9)
15. TCEPH-Tris(2-chloroethyl) phosphate (8)
16. DEHAZ-Di(2-ethyl hexyl) azelate (6)
17. TPP-Triphenyl phosphate (6)
18. DTAMQ-2,5 Ditert amyl quinone (5)
19. PCB-Polychlorinated biphenyls (5)
20. DEHAD-Di(2-ethyl hexyl) adipate (4)

2010 Top 20

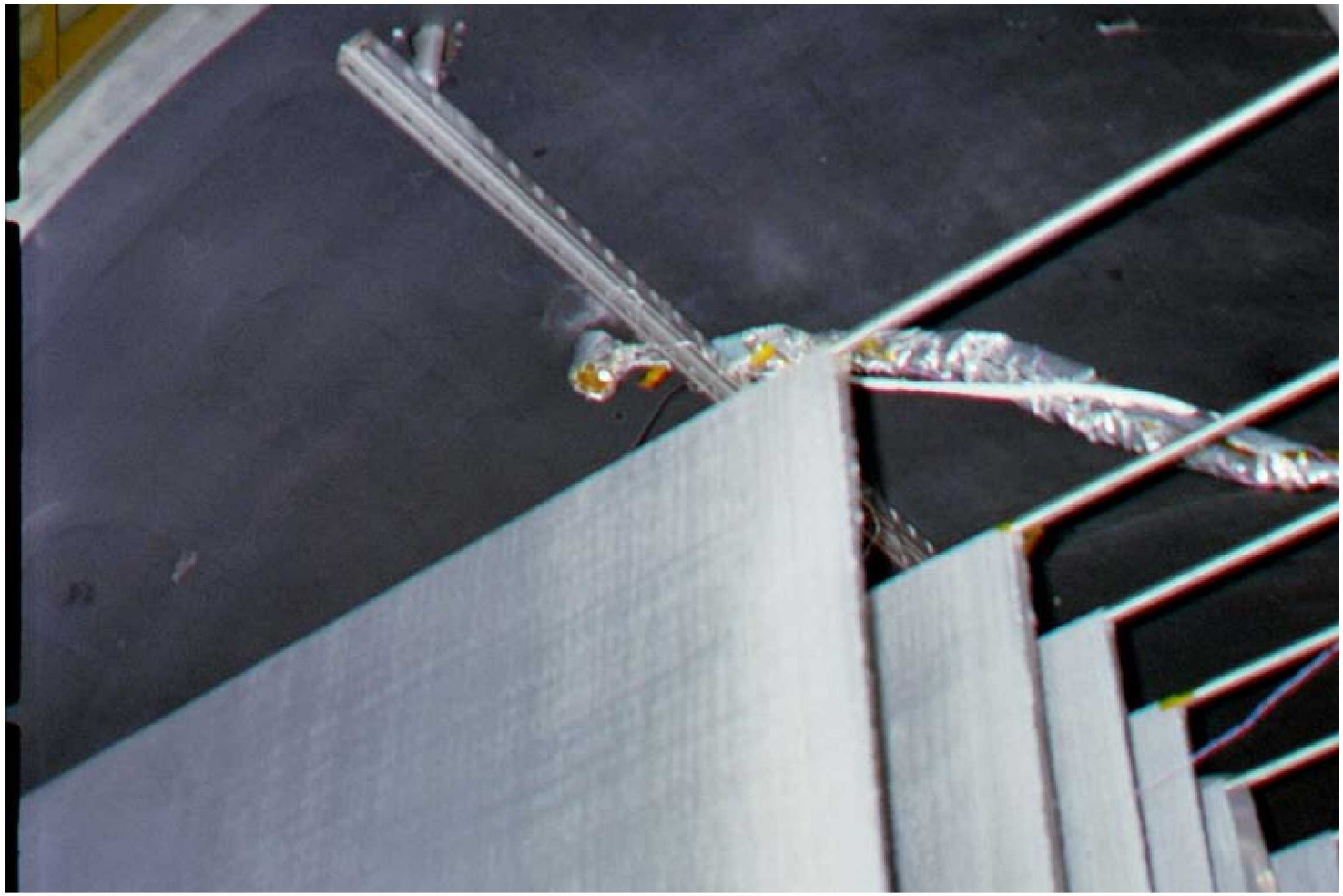
1. HYD-Various Hydrocarbons (151)
2. DEHP- Di (2-ethyl hexyl) phthalate-Plasticizers (135)
3. DBP-Dibutyl Phthalate- Plasticizers (93)
4. MESIL-Methyl Silicones (74)
5. ORGAC-Organic acids-surfactants (62)
6. DEHAD-Di(2-ethyl hexyl) adipate-plasticizer (46)
7. CHAF-Cyclohexylamine or formamide -boiler steam additives (44)
8. ALLYL-Tris(allyl) cyanurate(TAC)- wire insulation (40)
9. ESTER-Esters (38)
10. BNZE-Benzoate esters –plasticizers (36)
11. RTV56-Methyl Phenyl Silicones (34)
12. TPP-Triphenyl Phosphate-fire retardant (25)
13. BNZA-Benzoic acids-surfactants (24)
14. DINO-Dinonyl phthalates- Plasticizers (23)
15. BTC-Butyl Compounds-detergents (21)
16. PHEN-Phenols-antioxidants (19)
17. DEHS-Di(2-ethyl hexyl) sebacate (19)
18. OCTE-Octanoate Esters-plasticizers (18)
19. BTYLP-Butyl palmitate-surfactants (18)
20. ACRE-Acrylate ester-cable contaminant (16)

Common species -MLI

MLI-Germanium Black Kapton, Kapton, VDA mylar layups with polyester scrim, finished w/thread and velcro &acrylic-backed tapes

- DEHP-vinyl plasticizer
- Adipate esters- plasticizer, from thermal blankets
- Organic Acids- from fingerprints and cottonseeds
- Various hydrocarbons-greases from threading, transfer contaminant
- Isophthalate esters- from thermal blankets

MLI TV Bakeout

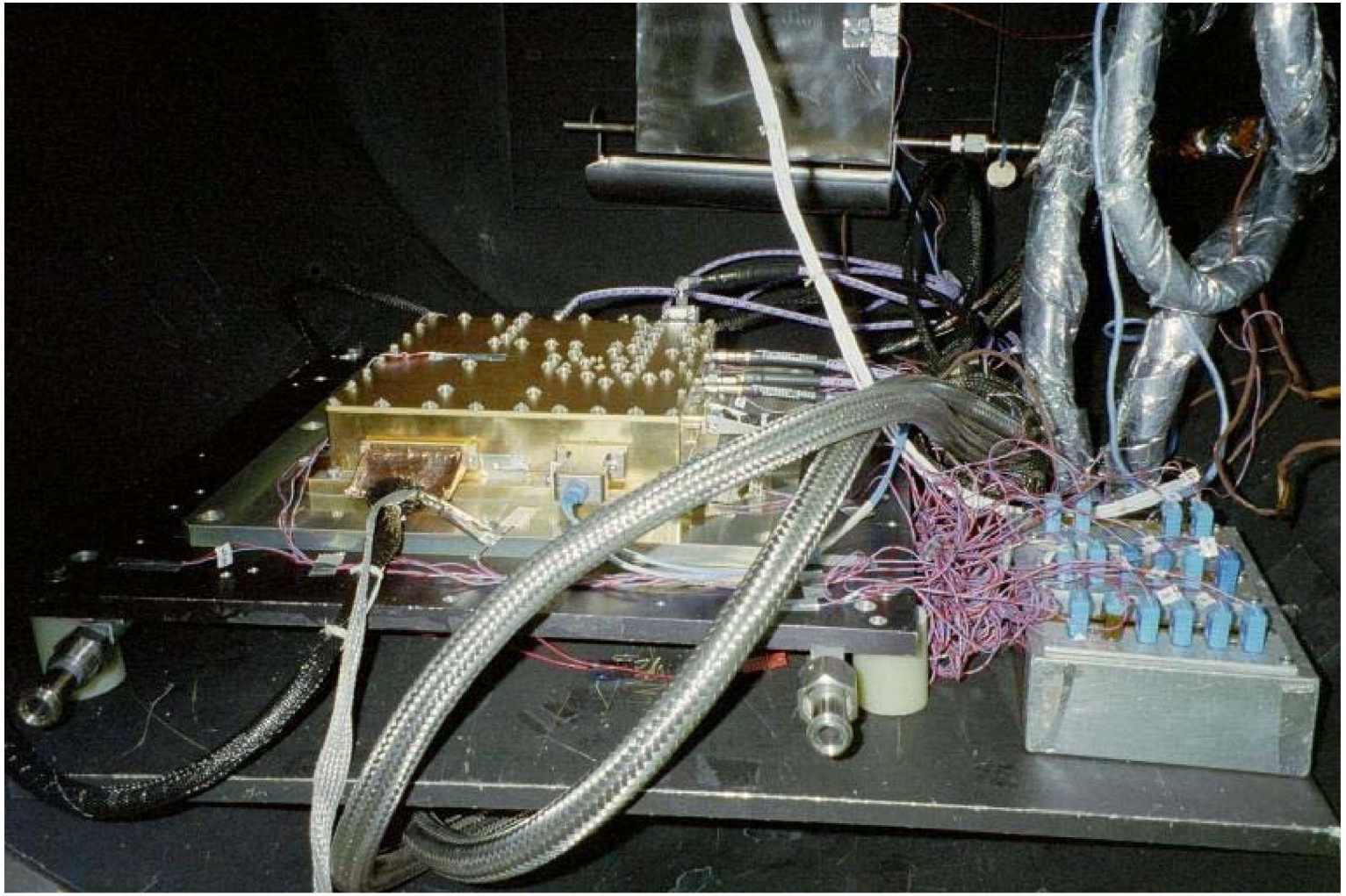


Common species -Structures

Graphite epoxy structural panels (GrEp on Al honeycomb), tubes, booms for HGA, film adhesives, bonding epoxies

- DEHP-vinyl plasticizers
- Various hydrocarbons-greases, oils, byproducts
- DBP-plasticizers
- TPP-flame retardant
- Bisphenol-A and amine based curing agents- from epoxies

Electronic Box TV

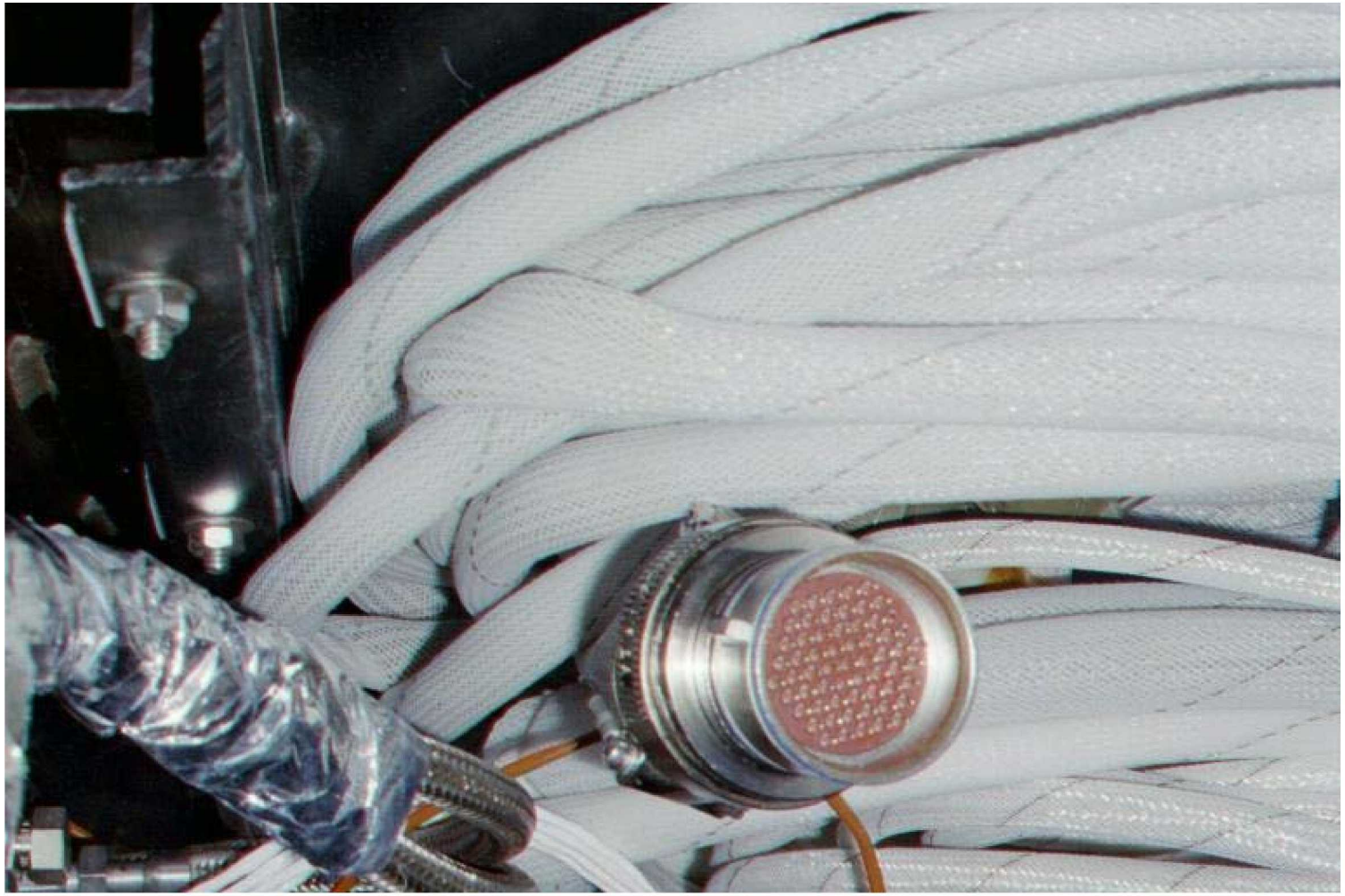


Common species – Electronic units

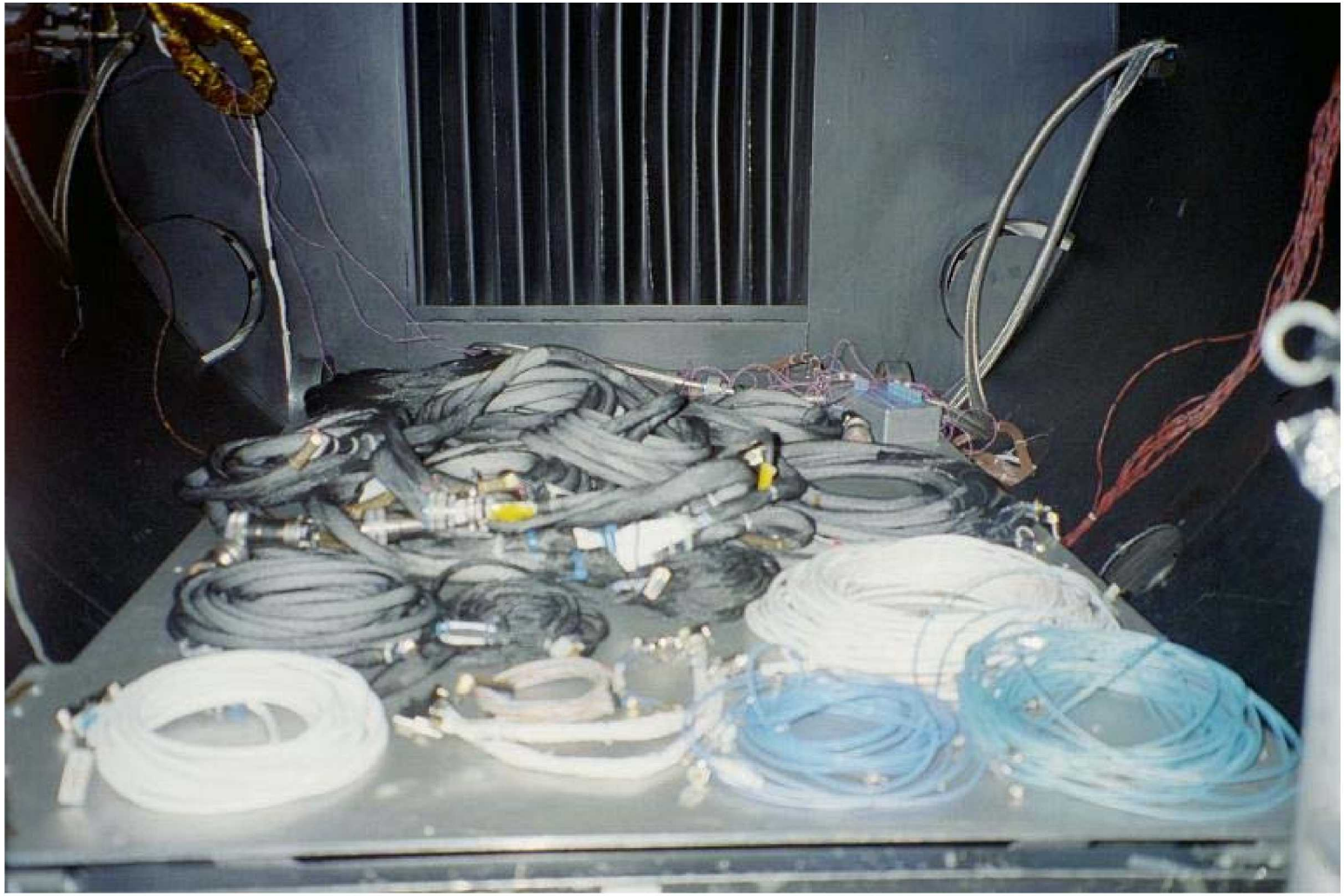
Electronic boxes, PWBs, thermal padding, harnesses

- DEHP-vinyl plasticizers, light oils
- Various hydrocarbons-greases, machining oils, byproducts
- DBP-plasticizers, paint byproducts
- TPP-flame retardant, paint byproducts
- Bisphenol-A and amine based curing agents- from epoxies
- Methyl Phenyl silicones- elastomers on connectors and thermal padding (Choseal, chotherm, CV products)
- Aromatic Amides- from PWBs and electronics

Harness Bakeout



Harness Bakeout



Common species – Harnesses,cables

GSE & Flight harnesses and cables

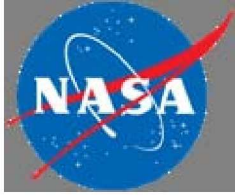
- Various hydrocarbons-greases, machining oils, byproducts
- DEHP-vinyl plasticizers, light oils
- Tri (allyl) cyanurate (TAC) cross-linking agents from wire insulation of Tefzel cables
- Organic Acids-handling contaminants
- Fatty acid esters-surfactants
- Phenol-based compounds-antioxidants (prevent oxidation)
- Urethanes-from encapsulants, potting
- Methyl silicones-mold releases, elastomers, greases

Unique species

From hardware, airborne

- Triphenyl Phosphates- flame retardant plasticizer
- Cyclohexylamine (CHA)- boiler steam/anti-scaling additive, airborne
- Hexa (methoxymethyl) melamine (HMMA)-resins from calrods taken over 300C.
- Butoxy (or ethoxy) compounds –detergents, used to clean GSE, hardware
- Squalene- from skin oil, handling/transfer contaminant
- Caprolactum- bagging contaminant from nylon products

Methods to Reduce NVR



- Review Materials list for flight and GSE hardware.
- Pre clean your hardware with approved cleanroom supplies.
  - Precision clean, NVR sample, possibly reclean, resample.
- Visually inspect critical hardware with white and black light.
- Bake hardware at as high of a temperature as allowed, as long as feasible.
- Store cleaned hardware in low outgassing bagging material (ULO polyethylene or Llumalloy).
- Use red non-flight covers whenever possible.
- Handle hardware with low NVR gloves (Nitrile or polyethylene)
- Store cleaned and baked out hardware in cleanrooms or GN2 purged dry boxes.
- Ship cleaned and baked out hardware doubled bagged and purged with dry filtered air or GN2.

Acknowledgements

- Special appreciation goes to Joe Colony and Fred Gross who produced the chemical analysis methodologies and early literature that covered spacecraft materials.
- Special thanks goes to the personnel over the years from the GSFC Materials Branch whom have honed the standards for chemical analytical testing at GSFC.
- Thanks to the NASA chemists, Doris Jallice and Alex Montoya, who do a lion's share of the analytical identification of chemical species and produce excellent and insightful information in the reports.
- Thanks to the personnel that have worked and managed the operation of the GSFC TV chambers in Building 7 (Ed Packard, Leon Tilwick, Mike Bullinger, & Mike Schools).
- Co-author, Tony, for his review of material contained within the paper and presentation.
  - Refer to his tutorial presented herein at the 26th SSC.